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NATIONAL BUREAU OF STANDARDS REPORT

9997

MICROPLASTICITY II, MICROSTRAIN BEHAVIOR OF NORMALIZED 4340 STEEL AND ANNEALED INVAR

To

Materials and Processing Branch
Naval Air Systems Command
Department of the Navy

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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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Microplasticity II, Microstrain Behavior of Normalized 4340 Steel and Annealed Invar

G. W. Geil and I. J. Feinberg

The testing techniques and some precautions necessary for obtaining accurate measurement of small microstrains were discussed in a previous

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report (1). The results obtained in a study of the microplastic behavior

ES FeB

of normalized 4340 steel and annealed Invar at 24.20 C ± 0.01 C are presented in this report.

The vacuum melted 4340 steel had the following chemical composition, in percent by weight: carbon 0.40; manganese 0.68; phosphorus <0.01; sulphur <0.01; silicon 0.33; nickel 1.80; chromium 0.79; molybdenum 0.24;

ES

balance iron. The normalizing treatment consisted of holding the sample at 900 C (1650 F) for one hour followed by air cooling.

The Invar sample had the following chemical composition in percent by weight: carbon 0.14, manganese 0.50, nickel 34.31; iron 64.64, sulphur 0.004, phosphorus 0.002, and a 0.4 balance of silicon, chromium, copper

FeB

and cobalt. It was given the annealing treatment, usually prescribed, of holding at 830 C (1525 F) for one hour followed by quenching in water.

ES FeB

Tensile and microplasticity specimens were prepared from the 4340 steel and Invar samples after heat treatments. They were finished by careful grinding to their final dimensions. → p 13

The designs of the microplasticity specimens and the capacitance gage assembly of three gages spaced 120° apart around the specimen and the testing procedures used in these microplasticity studies are described in the previous paper (1). The temperature of the insulated thermal

chamber surrounding the specimen and capacitance gage assembly was maintained at 24.20 C ± 0.01 C. The microplasticity tests were conducted using a step loading-unloading procedure, progressively loading to higher stresses. Microstrains of the 2-inch gage length of the specimens were determined to within ± 1×10^{-7} from readings of the three gages, except readings of one gage only for the intervals of less than one minute after unloading. The residual microstrains were determined from the initial gage readings taken prior to stressing the specimen and readings taken at zero stress after complete unloading of the specimen in each of the loading-unloading steps.

RESULTS AND DISCUSSION

<u>Normalized 4340 Steel</u>. The temperature of a specimen of normalized 4340 steel changes during a microplasticity test; cooling of the specimen occurs during loading within its elastic range, and heating occurs during any plastic deformation and during unloading (1,2). An interval of 10 to 15 minutes was usually required for the specimen to regain its initially controlled temperature (1). Thus a waiting period of 10 or more minutes prior to taking microstrain readings after unloading of the specimen was required for the exclusion of any thermal strains resulting from the temperature changes of the specimen.

Some of the data obtained in microplasticity tests on a single specimen of normalized 4340 steel are presented in Fig. 1. The step loading - unloading stages were conducted at a rate of approximately 50 ksi/min (5.8 MN/m^2/s). For clarity of presentation of the deformation behavior at small microstrains, only data for deformation up to microstrains of

6×10^{-6} are presented; peak stresses (maximum stress of each loading - unloading stage) are plotted versus the residual microstrains determined at zero loads. The microplastic yield stress, (hereinafter) designated as "MYS" in this report, is the stress value taken from the peak stress - residual strain curve at a residual strain of 1×10^{-6}.

The peak stress-residual strain relationship obtained in the initial microplasticity test of the specimen (Fig. 1, curve A) indicates a MYS of 37.5 ksi (258 MN/m^2). This stress is approximately one-fourth of the 151 ksi (1040 MN/m^2) yield strength (0.2% offset) of the normalized 4340 steel specimen. No appreciable residual strains at zero stress were observed in this test after unloading from peak stresses below 30 ksi (207 MN/m^2). This test was discontinued after loading to a peak stress of 50 ksi (345 MN/m^2) with a residual strain of 4.8×10^{-6} measured 20 minutes after complete unloading of the specimen. The portion of this curve designated by "R" represents the measured recovery of residual strain (contraction of the specimen) of about 0.8×10^{-6} that occurred under zero stress during an interval of 40 hours. This recovery feature will be discussed in some detail later.

The influence of pre-microstraining of the specimen on its subsequent peak stress-residual strain relationships is depicted by the data presented in curves B to G of Fig. 1. The maximum peak stress of the preceding microplasticity test and the pre-microstrains of the specimen are reported in the legend for the figure. The MYS values of 58 ksi (400 MN/m^2) and 59 ksi (407 MN/m^2) for the specimen after small total pre-microstrains of

4×10^{-6} and 28×10^{-6} (curves B and C, respectively) are significantly higher than the 37.5 ksi (258 MN/m^2) obtained in the initial microplasticity test. This increase in MYS values may be attributed mainly to some work hardening of the specimen during the previous microstraining and perhaps to some redistribution of initial residual stresses in the specimen.

Three pronounced instability characteristics that were introduced into the specimen by microstraining were revealed by these microplasticity tests. These are (1) considerable recovery of plastic microstrain at zero stress during an interval varying from less than one hour to one or more days after complete unloading of the specimen, (2) appreciable microstraining at very low stresses during reloading of the specimen when there had been recovery of plastic strain after the previous test; and (3) reversals in the peak stress-residual strain relationships.

(1) Recovery of plastic microstrain at zero stress.

Some recovery of plastic microstrain at zero stress in an interval varying from less than one hour to several hours on days after unloading was observed in all tests, even in the initial test which involved a total microstrain of only 4.8×10^{-6}. The total strain recovery generally increased with increase in the microstrain during the test and especially with increase in the total pre-microstrain of the specimen. This latter feature is illustrated by the recovery strains, "R", observed in the test on the specimen after a total pre-microstrain of 1.57×10^{-3} (Fig. 1, curve G). The intervals between the recovery measurements are indicated. The first measurement of residual microstrain

(2.4×10^{-6}) was made 20 minutes after complete unloading of the specimen from the maximum peak stress of the test, so as to eliminate any inclusion of thermal strains; an appreciably recovery of plastic microstrain may have occurred in this interval. An increase in the strain recovery was observed with increase in time up to 2 days. No additional recovery was observed with further increase in time. Practically all of the microstrain of the specimen in this microplasticity test was recovered.

(2) Microstrains at low stress levels after pre-strain and recovery.

In all of the microplasticity tests on the pre-microstrained specimen small microstrains were observed at peak stresses very much lower than the MYS of the specimen, or the maximum peak stress of the previous microplasticity test. These microstrains at low peak stresses did not depend greatly upon the magnitude of the prestraining in the previous tests up to total pre-microstrains of 460×10^{-6} (curves B, C, D and E, Fig. 1). However, with much greater total prestrains of the specimen, such as 1.56×10^{-3} or 1.57×10^{-3}, the microstrains at low peak stresses during the subsequent microplasticity test were considerably larger (curves F and G, Fig. 1), reaching a value of 1×10^{-6} at peak stresses slightly over 40 ksi (280 MN/m^2).

These microstrains at low peak stresses apparently are directly related to the strain recovery after the preceding microplasticity test. It is tentatively suggested that during the period of observed strain recovery edge dislocations move backward, as opposed to their forward motion under applied stress, perhaps by a double kink mechanism under the internal back stresses in the specimen after unloading. It is assumed

that on restressing the specimen in a subsequent microplasticity test the forward movement of the edge dislocations by a double kink mechanism towards their positions prior to the recovery is facilitated somehow as a result of their previous movements.

(3) Reversals in the peak stress-residual strain relationships.

Reversals in the peak stress-residual strain curves at peak stresses ranging from 50 to 70 ksi (350 to 480 MN/m^2) are present (Fig. 1, curves D,E,F and G) for microplasticity tests conducted after previous tests to residual strains of 208 x 10^{-6} and higher. These reversals with accompanying small decreases in microstrain (contraction of the specimen) occurred following peak stresses much smaller than the highest applied peak stress of the previous microplasticity test. A trend of a decrease in the peak stress at reversal with increase in the pre-microstrain of the specimen is indicated in Fig. 1 by the approximately 10 ksi (70 MN/m^2) lower peak stresses for the reversals in curves F and G than those in curves D and E.

Normalized 4340 Steel Prestressed to 1.5 Percent Extension. The usual practice in many microplasticity investigations is to prestrain specimens, extending them within a range of 0.1 to 5 percent before conducting microplasticity tests (3). In order to study the effect of prestraining within the above range on the behavior of the normalized 4340 steel specimen, it was stressed to 225 ksi (1550 MN/m^2) with a resulting total plastic extension of 1.5 percent. Microplasticity testing of the specimen was delayed for 20 days, a period longer than that believed necessary for completion of all recovery features. The data obtained in the microplasticity

test conducted at a loading-unloading rate of approximately 100 ksi/min (11.5 $MN/m^2/s$) are presented in Figs. 2, 3 and 4.

The insert in Fig. 2 presents the observed peak stress-residual strain relationship to a microstrain of 3×10^{-6} using an expanded abscissa scale similar to the one in Fig. 1. A very small, probably insignificant, positive residual microstrain at zero stress was measured after step loading to a peak stress of 6 ksi (41 MN/m^2). However, after further step loading-unloading stages to peak stresses of 16 ksi (110 MN/m^2) and 28.5 ksi (196 MN/m^2), negative residual microstrains (contractions) of 3.5×10^{-6} and 7.5×10^{-7}, respectively, were observed. Positive residual microstrains were again observed after step loading to successively higher peak stresses. The reversal in the peak stress-residual strain curve of this test occurred at a peak stress between 6 ksi (41 MN/m^2) and 16 ksi (110 MN/m^2). This peak stress value is much lower than the peak stress values at the reversals shown in Fig. 1, and confirms the previously mentioned, general trend of a decrease in peak stress at the reversal in the peak stress-residual strain relationships with increase in the prestrain of the specimen. An adequate explanation is not readily available as to the involved dislocation mechanisms or possible phase and microstructural changes in the specimen that produce the observed reversals in the peak stress-residual strain relations and the relatively small residual contractions of the specimen on step loading-unloading to peak stresses just above the peak stress value at the reversal.

The observed contraction of the specimen and reversal in the peak stress-residual strain curve (Insert of Fig. 2) for peak stresses below 44 ksi (303 MN/m^2) reveal that the specimen was still dimensionally unstable even 20 days after the prestrain treatment. Moreover, some microstrain recovery was observed after unloading of the specimen from peak stresses of 28.5 ksi (196 MN/m^2) and higher. This is shown in Fig. 2 by the differences between residual microstrain measurements, curve A, taken 10 to 20 minutes after unloading (interval necessary to eliminate any inclusions of thermal strains) and microstrain measurements, curve B, taken approximately 1 hour after unloading. Furthermore, the two residual strain measurements, curve C, taken 17 hours after unloading from the peak stresses of 113 ksi (770 MN/m^2) and 157 ksi (1080 MN/m^2) indicated additional microstrain recovery in the interval between approximately 1 hour and 17 hours. Some microstrain recovery of about 7×10^{-7} was observed even during the interval between measurements taken 17 and 41 hours after unloading from the peak stress of 157 ksi (1080 MN/m^2).

To obtain further information on recovery behavior and thermal strains, the specimen was stressed to a peak stress of 164 ksi (1130 MN/m^2). The temperature changes of the specimen during loading, unloading, and at zero stress after unloading were determined from milli-microvoltmeter readings of a Chromel-Alumel thermocouple taped tightly to the surface of the specimen at mid-point of the gage length section. The temperature data, (Fig. 3) reveal a relatively large decrease of over 0.6 C during loading of the specimen and a rise to about 0.2 C above its initial temperature after complete unloading.

The first microstrain reading was obtained 15 seconds after complete unloading of the specimen. The data points in Fig. 2 for the instant of complete unloading (zero time) were determined by extrapolation of an expanded residual strain versus time curve (not shown) extending over an interval of five minutes. The thermal strain portion of the total microstrain after unloading is based upon the temperature changes of the specimen after unloading (Fig. 3) and a value of 11.3×10^{-6} in./in./C (m/m/C) for the coefficient of linear thermal expansion of 4340 steel at ambient temperature.

The time relationships for total contraction, plastic contraction and thermal contraction of the 4340 specimen at zero load after step loading to the peak stress of 164 ksi (1130 MN/m^2) and unloading (Fig. 2) are presented in Fig. 4. Most of the thermal contraction occurred during the first 10 minutes. Plastic contraction was very rapid at first and decreased at a continuously decreasing rate over an interval of 648 hours (27 days). There was a total recovery of plastic contraction of 20.8×10^{-6} in./in. which is approximately two-thirds of the residual plastic strain, 32.5×10^{-6} (Fig. 2), generated during the step loading-unloading stage of the 164 ksi (1130 MN/m^2) peak stress.

The microplasticity data presented in Figs. 1, 2 and 4 clearly reveal the high degree of instability of the prestrained specimen of normalized 4340 steel and the very long intervals required for completion of the recovery processes. A general trend is also shown of an increase in instability and in recovery interval with increase in the prestrain of the specimen.

Additional studies are planned to determine if the instability behavior of the normalized 4340 steel is characteristic of that in other steels and in 4340 steel with other microstructures resulting from different heat treatments.

Annealed Invar. A microplasticity test specimen was prepared from the sample of annealed Invar. It was finished by careful grinding to its final dimensions. Peak stress-residual microstrain data obtained in microplasticity test with loading-unloading steps conducted at a rate of 10 ksi/min (1.1 $MN/m^2/s$) are presented in Fig. 5. The residual microstrain measurements at zero stress were made at various intervals, ranging from 1 to 135 minutes after unloading the specimen from the indicated peak stresses. The step loading-unloading and residual strain measurements were conducted to a maximum peak stress of 15 ksi (103 MN/m^2) with an accompanying residual microstrain of 15×10^{-6}. No microstrain recovery features, such as those observed with the normalized 4340 steel, or other instability effects were observed in this test; the residual microstrain measurements after unloading from each peak stress were independent of time.

Due to the very low coefficient of linear thermal expansion at ambient temperature of the specimen of annealed Invar, its temperature changes during loading and unloading within its elastic range were very small, less than 0.01 C (1). The specimen regained its initial temperature within one minute after complete unloading. Thus, the observed residual microstrain measurements made at intervals ranging from 1 to 135 minutes after unloading did not include any appreciable thermal strains.

The data presented in Fig. 5 indicate a MYS of 6.3 ksi (43 MN/m^2) for the annealed Invar. This stress value is about 15 percent of the determined 43.1 ksi (297 MN/m^2) yield strength (0.2% offset) of the annealed specimen.

Annealed Invar Prestrained to 1.94 Percent Extension. The specimen of annealed Invar was stressed to 50.4 ksi (347 MN/m^2), with an accompanying extension of 1.94 percent, to evaluate the effects of prestraining on its subsequent microplastic behavior. Microplasticity testing was delayed several days to provide sufficient time for any recovery of strain that may have occurred after prestraining.

The data obtained in the tests conducted 8 and 9 days later on this specimen are presented in Fig. 6. Prestraining to 1.94 percent extension introduced a high degree of instability in the Invar specimen. This is revealed in the strain recovery (contraction of the specimen) at zero stress after each of the step loading-unloading stages up to a peak stress of 24 ksi (165 MN/m^2). The residual microstrain values were independent of the interval between unloading and strain measurements in the tests up to 15 ksi (103 MN/m^2) peak stresses and only slightly dependent on the interval after unloading from peak stresses between 15 ksi (103 MN/m^2) and 24 ksi (165 MN/m^2). However, after the loading-unloading steps to peak stresses greater than 24 ksi (165 MN/m^2), some initial extension of the specimen followed by appreciably recovery of residual strain with increase in time after unloading was observed. Moreover, this recovery generally increased with increase in the microstrain that occurred during the step loading-unloading stages. For example, most of the residual

microstrain that occurred during the loading step to a peak stress of 39 ksi (270 MN/m^2) and unloading was recovered within 20 hours after unloading the specimen. No additional recovery of microstrain was observed during the next 100 hours. Approximately two thirds of the total recovery of microstrain occurred during the interval of one hour after specimen unloading.

Any designation of a specific MYS value for this prestrained specimen of Invar is certainly questionable as it depends greatly upon the interval between specimen unloading and measurement of residual microstrain. For instance, a MYS of approximately 35 ksi (240 MN/m^2) is indicated (Fig. 6) by measurements taken 2 minutes after unloading, whereas, values of approximately 37 ksi (255 MN/m^2) and 39 ksi (270 MN/m^2), respectively, could be selected from the observed measurements at one hour and at 4 or more hours after unloading. These stresses are very much higher than the MYS of 6.3 ksi (43 MN/m^2) observed (Fig. 5) for this same specimen in its annealed condition.

The microplastic behavior of the specimen of normalized 4340 steel prestrained to an extension of 1.5 percent and that of the specimen of annealed Invar prestrained to an extension of 1.9 percent clearly demonstrate that prestraining introduces a high degree of instability; their microplastic behaviors were greatly different than those observed with the specimens in their initially heat-treated conditions. Thus, for these two materials and possibly for others, the somewhat general practice of prestraining the specimens to an extension of 0.1 to 5 percent prior to observing their microplastic behaviors does not provide desirable

information on the microplastic behavior of the materials in their initial conditions.

SUMMARY

The microplastic behavior of normalized 4340 steel subjected to tensile stresses at ambient temperature, 24.2 C (75.7 F), was investigated. A step loading-unloading procedure, progressively loading to higher peak stresses was employed. Residual microstrains of the 2-inch gage length of the specimen were determined at zero stress after each unloading stage, following the procedures described in the previous report, "Microplasticity I - Measurement of small microstrains at ambient temperature."

An initial microplastic yield stress (MYS) of 37.5 ksi at a strain of 1×10^{-6} was observed for the normalized 4340 steel specimen. Microstraining or prestraining the specimen introduced instability behavior during subsequent microplasticity tests. The following instability characteristics were observed: (1) significant recovery (contraction of specimen) occurred within intervals up to 24 hours or longer after complete unloading from peak stresses that produced appreciable plastic microstrains; (2) microstraining occurred at very low stresses on subsequent restressing if the specimen had undergone strain recovery following the previous plastic microstrain (these stresses being very much lower than the microplastic yield stress at 1×10^{-6} strain, or the maximum stress of the preceding test); (3) reversals were observed in the peak stress-residual strain curves at stresses below the maximum peak stress of the preceding test; and (4) prestraining to 1.5 percent extension, a procedure often employed in microplasticity investigations, enhanced rather than reduced instability.

Microplasticity tests were conducted on annealed Invar maintained at 24.2° C to a maximum peak stress of 15 ksi with an accompanying microstrain of 15×10^{-6}. No instability behavior such as that reported for the normalized 4340 steel, was observed. Moreover, no appreciable thermal microstrains were observed as the cooling of the specimen during loading within its elastic range and heating during unloading were very small due to the very low coefficient of linear thermal expansion of annealed Invar at ambient temperatures.

Prestraining of the annealed Invar specimen to an extension of 1.94 percent introduced instability characteristics similar to those observed with the 4340 steel.

The microplastic behavior of the specimen of normalized 4340 steel prestrained to 1.5 percent and that of the specimen of annealed Invar prestrained to 1.94 percent was greatly different than that observed with specimens of these alloys in their initial heat-treated conditions. Thus, for these two materials, and possibly for others, the often used practice of prestraining specimens up to 5 percent extension prior to observing their microplastic behaviors does not provide the desired information on the microplastic characteristics of the materials in their initial conditions. end

ACKNOWLEDGMENTS

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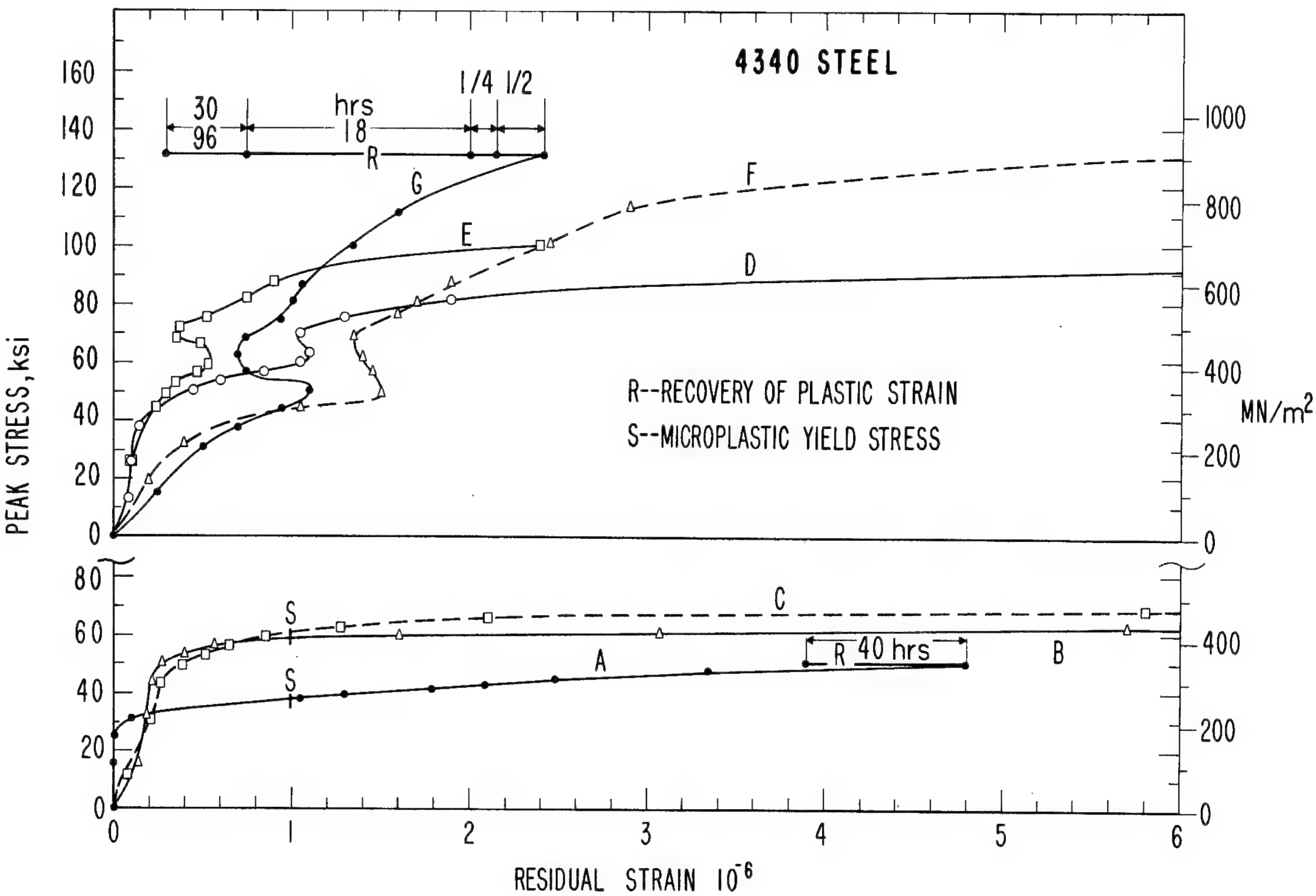

FIG. 1. Peak stress-residual strain relationships observed in microplasticity tests on a single specimen of normalized 4340 steel.

Curve	Maximum stress Previous test ksi	Maximum stress Previous test MN/m^2	Strain Previous test	Total Prior strain
A	0	0	0	0
B	50	345	4×10^{-6}	4×10^{-6}
C	69	480	24×10^{-6}	28×10^{-6}
D	94	650	181×10^{-6}	209×10^{-6}
E	110	760	19×10^{-6}	460×10^{-6}
F	148	1020	730×10^{-6}	1560×10^{-6}
G	138	950	8×10^{-6}	1568×10^{-6}

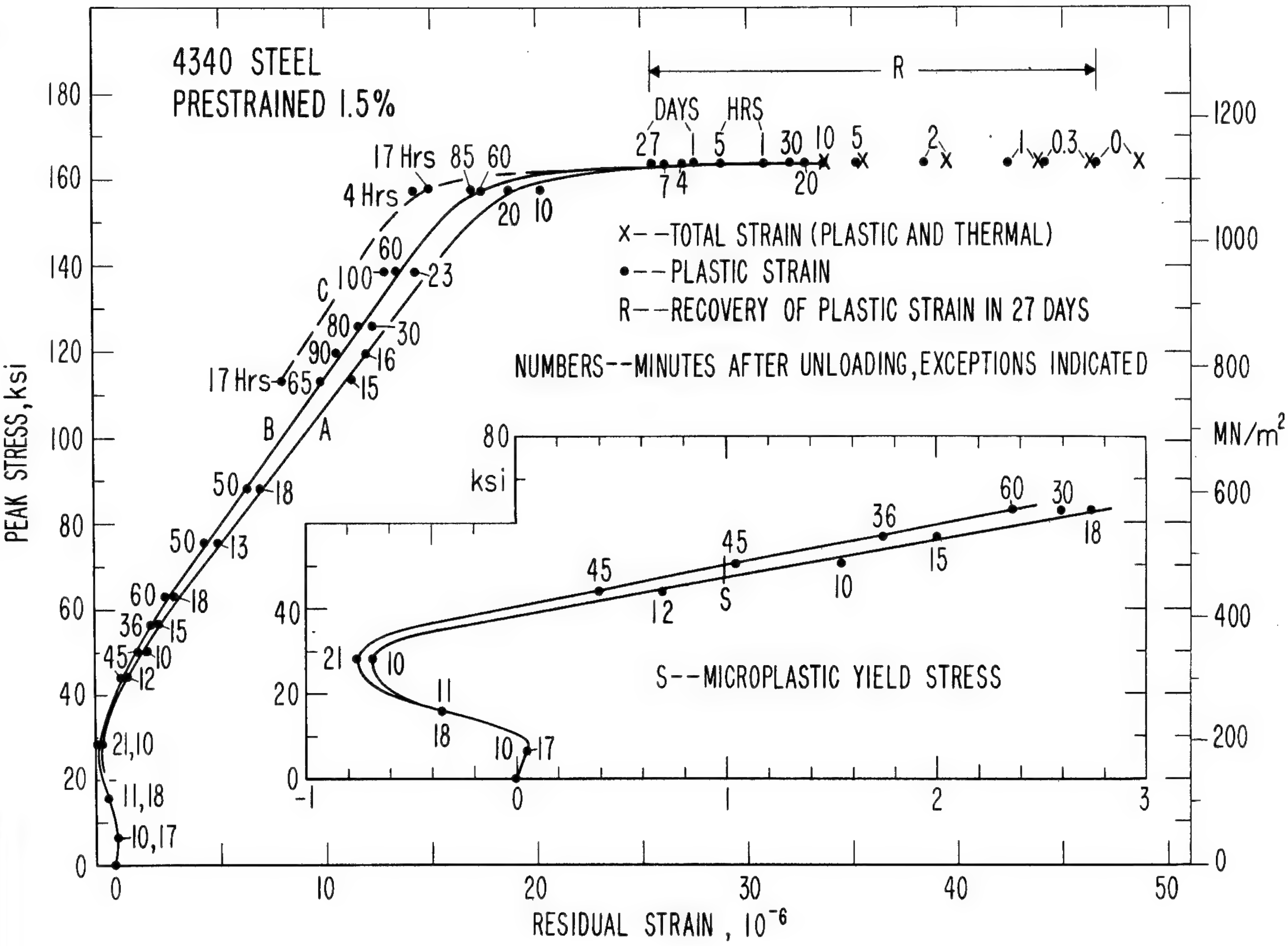

FIG. 2. Peak stress-residual strain relationships observed in microplasticity test on prestrained specimen of normalized 4340 steel. Specimen had been prestressed to 225 ksi (1550 MN/m^2) and extended 1.5 percent.

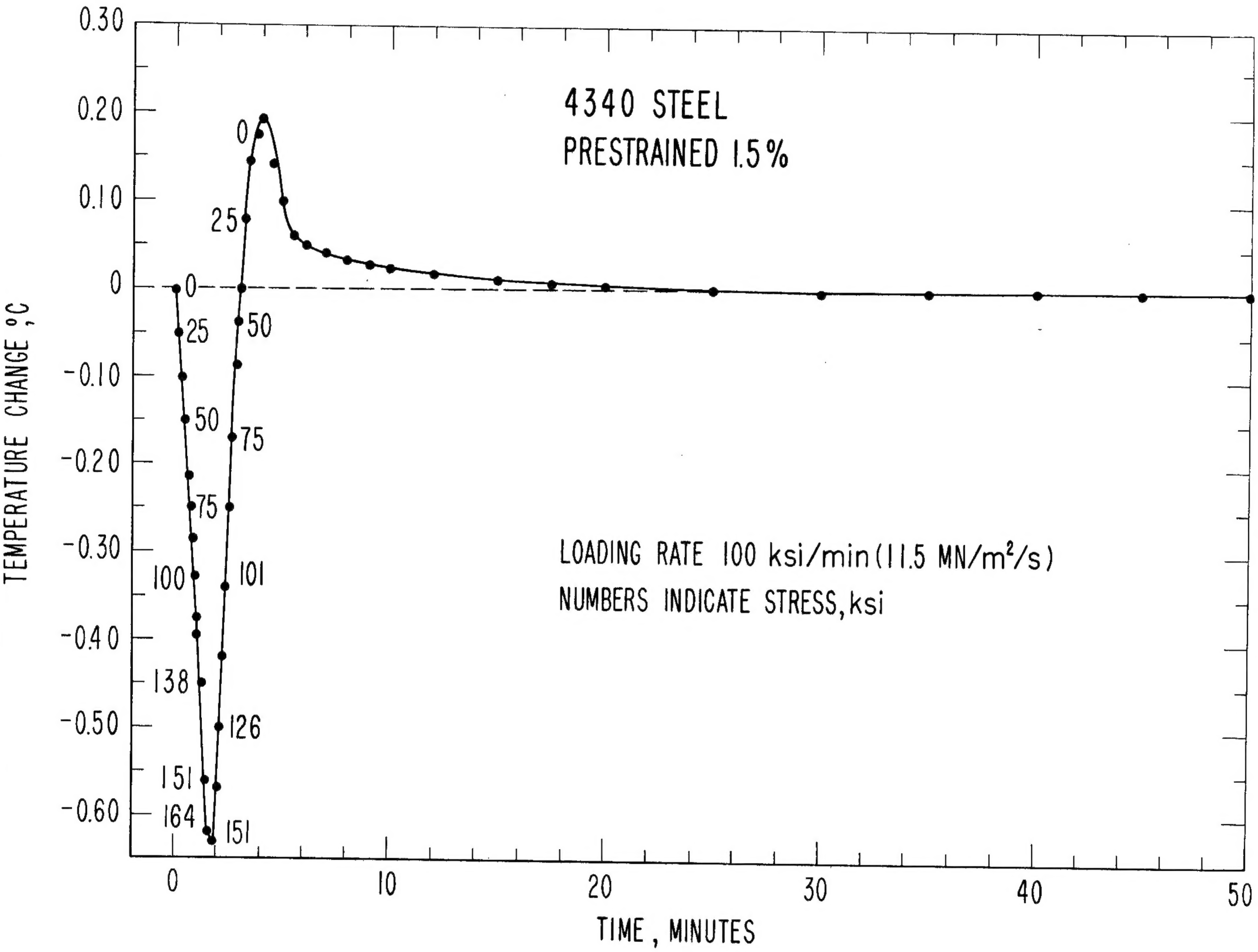

FIG. 3. Effect of loading at a rate of 100 ksi (11.5 MN/m^2/s) to a stress of 164 ksi (1130 MN/m^2) with immediate unloading, on the temperature change of the prestrained specimen of normalized 4340 steel. Specimen had been prestressed at 225 ksi (1550 MN/m^2) and extended 1.5 percent.

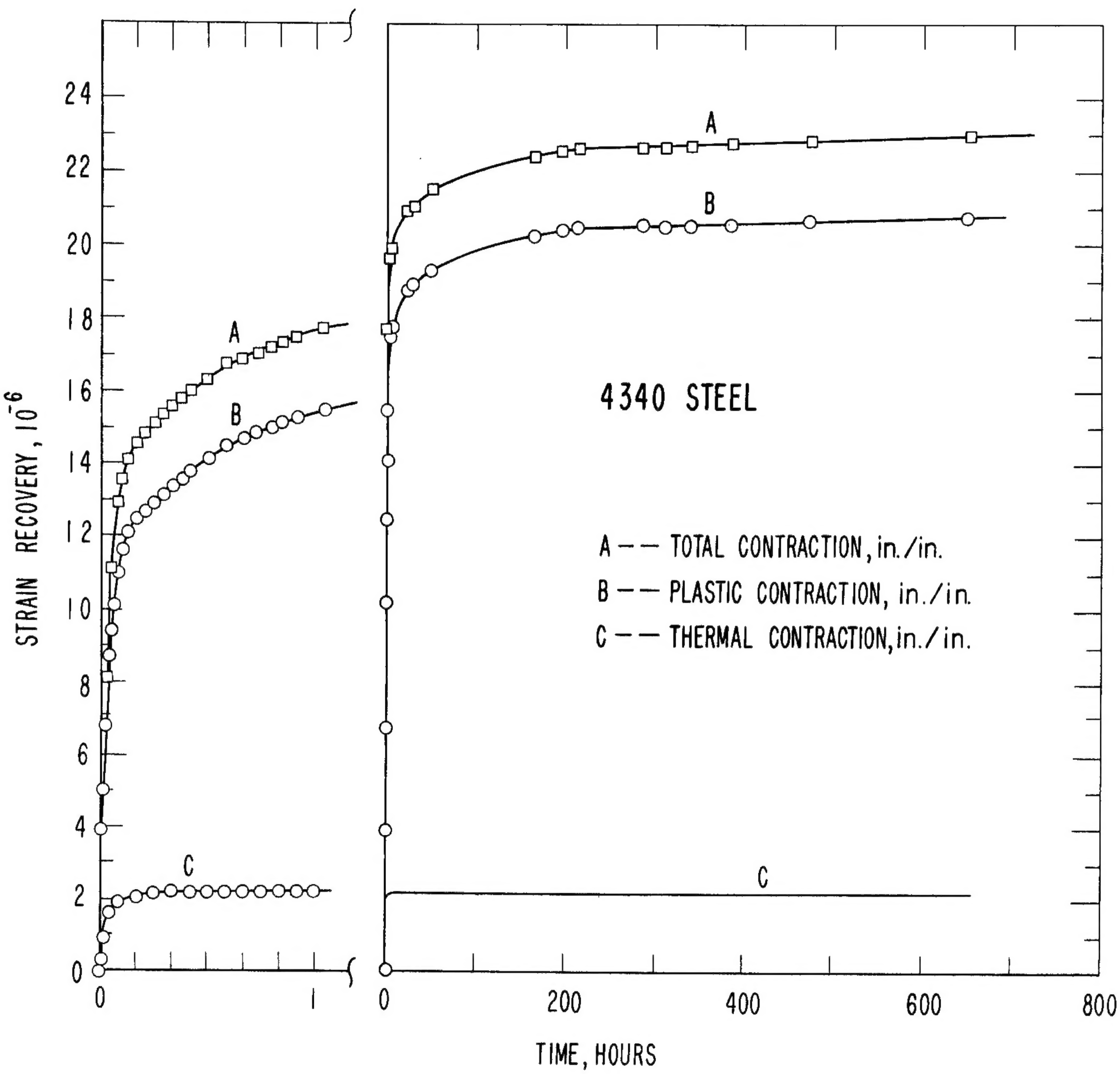

FIG. 4. Strain recovery-time relationships observed after unloading the prestrained specimen of normalized 4340 steel from the peak stress of 164 ksi (1130 MN/m^2). Specimen had been prestressed to 225 ksi (1550 MN/m^2) and extended 1.5 percent.

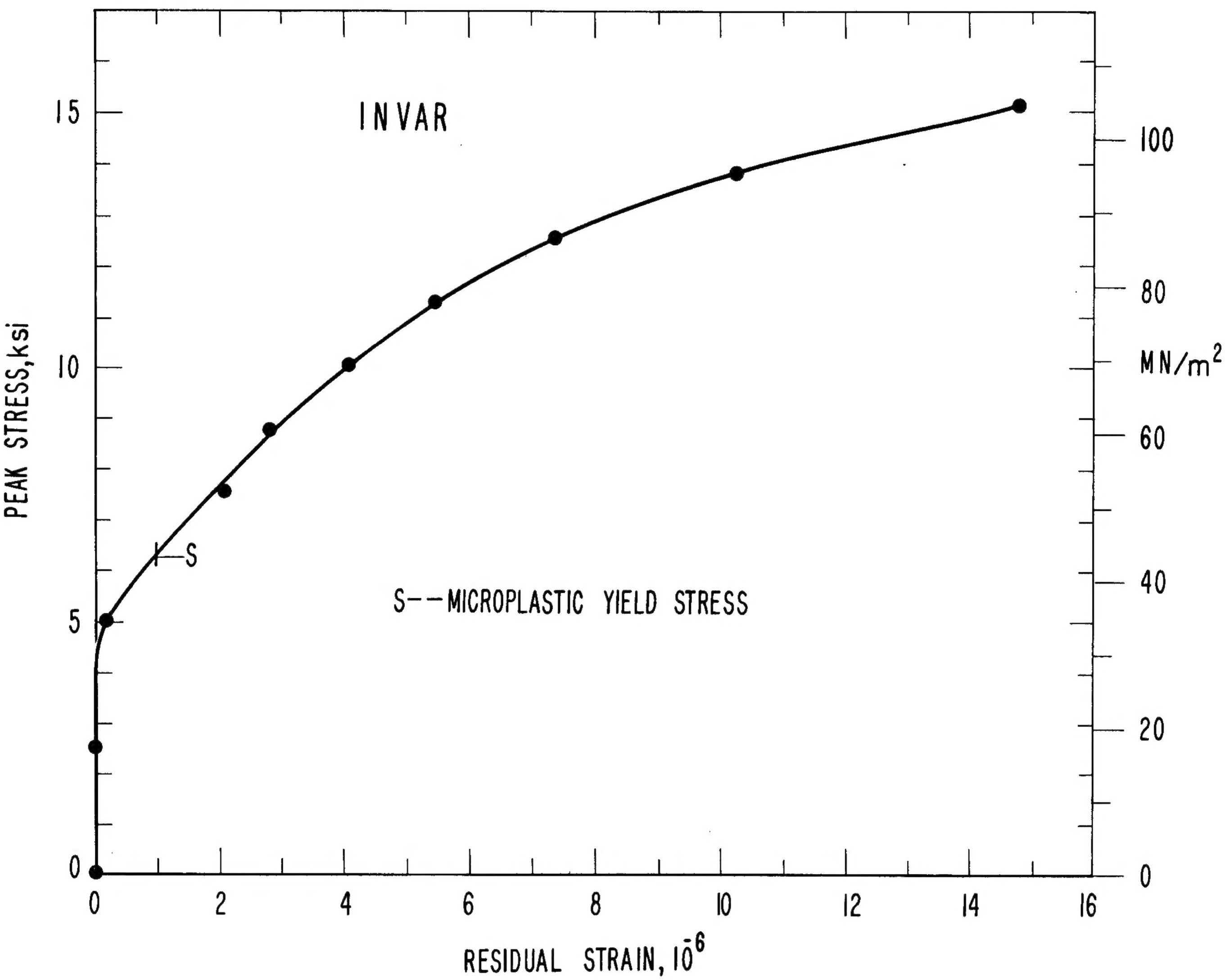

FIG. 5. Peak stress-residual strain relationship observed in microplasticity test on specimen of annealed Invar.

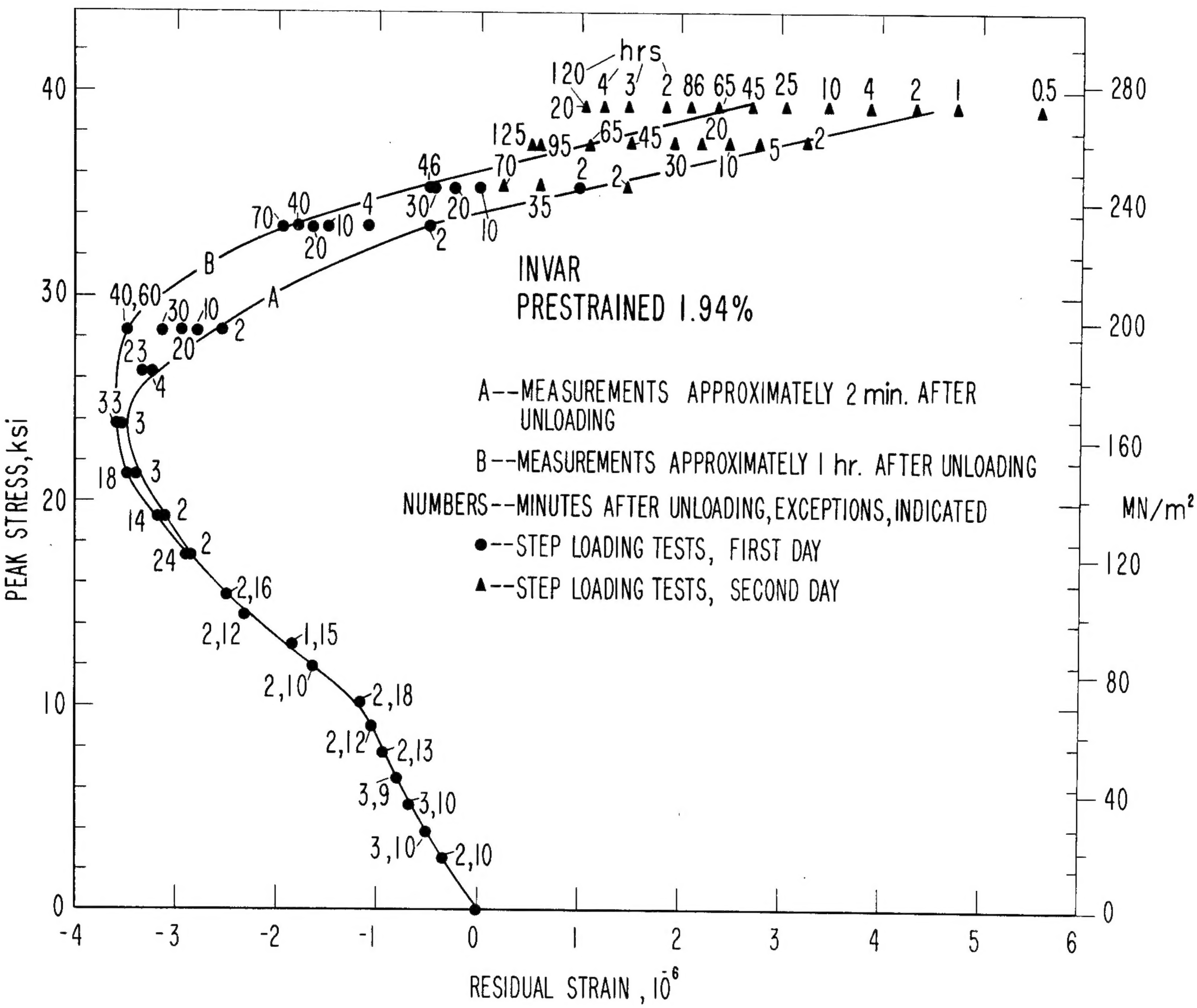

FIG. 6. Peak stress-residual strain relationships observed in microplasticity test on prestrained specimen of annealed Invar. Specimen had been prestressed to 50.4 ksi (350 MN/m^2) and extended to 1.9 percent.